

CHARACTERISATION AND MODELLING OF STATIC
RECOVERY PROCESS OF ALUMINUM ALLOY

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ABSTRACT

A comprehensive study of the annealing behavior of aluminum alloy has been carried out in this research. The primary objectives of this work have been to study the effect of recovery on the annealing behavior and to validate the static recovery process of aluminum alloy using Friedel's model. Annealing is a heat treatment process used to eliminate the residual stresses produced during cold work and static recovery is one of the processes in annealing. This process is the most subtle stage of annealing and no gross microstructure change occurred. In this research, aluminum alloy were to undergo thermo-mechanical process which compression test and annealing process took place. The experimental part has focused on characterisation of the softening reaction. Through this test, the material was tested at different pre-strain which is 2.5%, 5%, 7.5% and 10% to see the difference stress due to recovery temperature (100°C, 150°C, 200°C, 250°C) and time (1 hour, 2 hour, 3 hour, 4 hour). Then, the graph for relationship between degree of recovery, X_{rec} with temperature, time and pre-strain are plotted. Friedel's model equation is applied to the experimental result to find the activation energy, Q . For the relationship between degree of recovery, X_{rec} and temperature, the activation energy, Q is about 732.55 kJ/mol while the activation energy, Q for relationship between degree of recovery, X_{rec} and time is 224.83 kJ/mol. Finally, the activation energy, Q value from the experimental result are compared with literature review to validate the static recovery process of aluminum alloy using Friedel's model equation.

ABSTRAK

Pembelajaran menyeluruh tentang sifat sepuh lindap untuk aloi aluminium telah dilaksanakan di dalam kajian ini. Objektif utama kajian ini ialah mempelajari kesan pemulihan ke atas sifat sepuh lindap dan untuk mengesahkan proses pemulihan statik untuk aloi aluminium dengan menggunakan model Friedel. Sepuh lindap ialah pemulihan haba untuk menyingkirkan sisa tekanan yang terhasil dari kerja sejuk dan pemulihan statik ialah salah satu proses sepuh lindap. Proses ini merupakan peringkat yang paling halus di dalam sepuh lindap dan tiada perubahan struktur mikro yang kasar akan berlaku. Di dalam kajian ini, aloi aluminium akan melalui proses thermo-mekanikal iaitu ujian tekanan dan proses sepuh lindap. Bahagian eksperimental hanya tertumpu kepada ciri-ciri tindak balas kelembutan. Melalui ujian ini, bahan akan diuji dengan pra-ketegangan yang berbeza iaitu 2.5%, 5%, 7.5% dan 10% untuk melihat perbezaan tekanan dari segi pemulihan suhu (100°C, 150°C, 200°C, 250°C) dan masa (1jam, 2jam, 3jam, 4jam). Selepas itu, graf untuk hubungan darjah pemulihan, X dengan suhu, masa dan pra-ketegangan dihubungkan. Persamaan model Friedel digunakan ke atas hasil eksperimental untuk mendapatkan tenaga pengaktifan, Q . Untuk hubungan di antara darjah pemulihan, X dan suhu, nilai tenaga pengaktifan, Q ialah 732.55 kJ/mol manakala nilai tenaga pengaktifan, Q untuk hubungan antara darjah pemulihan, X dan masa ialah 224.83 kJ/mol. Akhirnya, nilai untuk tenaga pengaktifan, Q yang dikira daripada eksperimental akan dibandingkan dengan rujukan untuk mengesahkan proses pemulihan statik untuk aloi aluminium dengan menggunakan persamaan model Friedel.

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LIST OF SYMBOLS

X_{rec}	Degree of recovery
σ_{m}	Yield stress of the as deformed crystal
σ_{r}	Yield stress of the recovered crystal
σ_0	Yield stress of undeformed crystal
Q	Activation energy
R	Gas constant
T	Temperature
t	Time
C_1	Constant

LIST OF ABBREVIATIONS

Al	Aluminum
be	Back extrapolation
os	Offset
CNC	Computer numerical control
rpm	Revolution per minute

CHAPTER 1

INTRODUCTION

Annealing is one of the most important processes in industry. The term annealing refers to a heat treatment in which a material is exposed to an elevated temperature for an extended time period and then slowly cooled. Annealing is a heat treatment used to eliminate some or all of the effects of cold working. Ordinarily, annealing is carried out to relieve stresses and produce a specific microstructure. It also used to increase softness, ductility and toughness of metal.

Static recovery is one of the processes in annealing. The recovery temperature is heated just below the recrystallization temperature range. This process is the most subtle stage of annealing and no gross microstructure change occurs. During recovery, sufficient thermal energy is supplied to allow the dislocations to rearrange themselves into lower energy configurations. Recovery of many cold worked metals produces a subgrain structure with low angle grain boundaries and is this recovery process called polygonization. During recovery, the strength of a cold worked metal is reduced only slightly but its ductility is usually increased.

1.1 BACKGROUND

Aluminum alloys have found wide acceptance in engineering design primarily because they are relative lightweight and have a high strength to weight ratio. They also have a superior corrosion resistance and comparatively inexpensive. For some applications they are favored because of their high thermal and electrical conductivity, ease of fabrication and ready availability. Generally, the strengths of aluminum alloys decrease and toughness increases with increase in temperature and

with time at temperature above room temperature. Aluminum is easily fabricated. It can be cast by any method like rolled to any reasonable thickness, stamped, hammered, forged or extruded. It also readily turned, milled, bored or machined. Pure aluminum has low compression strength but when combined with thermo-mechanical processing aluminum alloy display a marked improvement in mechanical properties. The main application of aluminum is used in aircraft and rockets.

1.2 PROBLEM STATEMENTS

The problem statements are:

- (a) We want to find the different of stress before and after static recovery process.
- (b) Investigation of static recovery process due to influenced of pre strain, temperature and time.

1.3 OBJECTIVES

The research objective is to validate the static recovery process of aluminum alloy using Friedel's Model.

1.4 SCOPES OF PROJECT

- (a) In this research, the material used is limited to aluminum alloy only.
- (b) Test the specimen using compression test.
- (c) Varying pre-strain about 2.5%, 5%, 7.5% and 10%.
- (d) Perform the annealing using the box furnace.

1.5 METHODOLOGY

This research is conducted under several main steps. The major steps for this research are the material will go the compression test under a several pre-strain. Then, perform the annealing process to specimens using the box furnace. After that,

a few data are collect to perform the analysis. The Friedel's Model equation is using to validate the statistic recovery process for aluminum alloy.

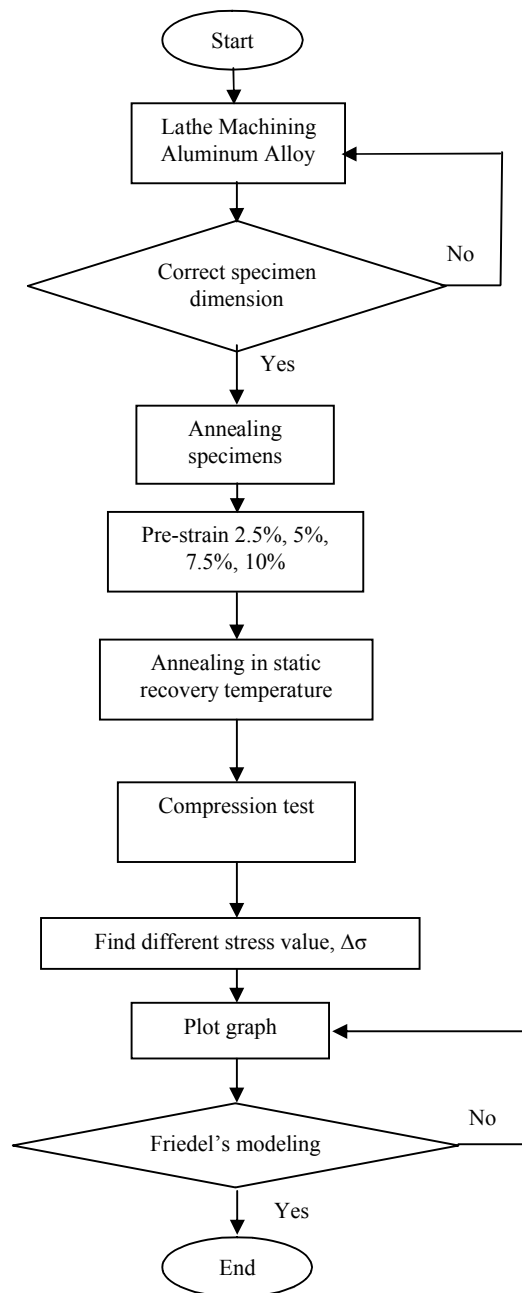


Figure 1.1: Flowchart of Methodology

CHAPTER 2

LITERATURE REVIEW

The literature review and background knowledge of static recovery process and how it related with Friedel's Model are presented in this chapter.

2.1 ANNEALING

Annealing is a heat treatment used to eliminate some or all of the effects of cold working. Annealing at low temperature maybe used to eliminate the residual stresses produced during cold working without affecting the mechanical properties of the finish part. Annealing also may be used to complete eliminate the strain hardening achieved during cold working. In this case, the final part is soft and ductile but still has a good surface finish and dimensional accuracy. After annealing, additional cold work could be done, since the ductility is restored by combining repeated cycles of cold working and annealing, large total deformations maybe achieved.

There are three stages in the annealing process, with the first being the recovery phase, which results in softening of the metal through removal of crystal defects (the primary type of which is the linear defect called a dislocation) and the internal stresses which they cause. The second phase is recrystallization, where new grains nucleate and grow to replace those deformed by internal stresses. If annealing is allowed to continue once recrystallization has been completed, grain growth will occur, in which the microstructure starts to coarsen and may cause the metal to have less than satisfactory mechanical properties. [1]

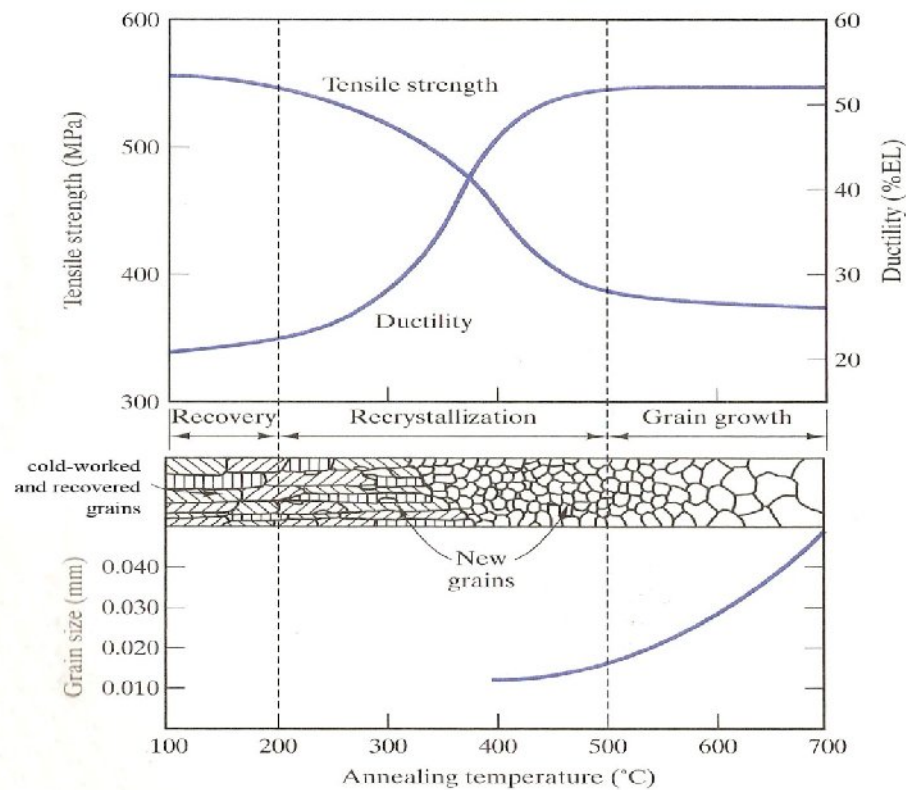


Figure 2.1: Effect of annealing on the structure and mechanical property changes.[3]

2.1.1 Recovery

Recovery is one of the process in annealing. The original cold-worked microstructure is composed of deformed grains containing a large number of tangled dislocations. When we first heat the metal, the additional thermal energy permits the dislocations to move and form the boundaries of a polygonized sub grain structure. The dislocation density however is virtually unchanged. This low temperature treatment removes the residual stresses due to cold working without causing a change in dislocation density and is called recovery. The mechanical properties of the metal are relatively unchanged because the number of dislocations is not reduced during recovery. However, since residual stresses are reduced or even eliminated when the dislocations are rearranged and is often called a stress relief anneals. [1]

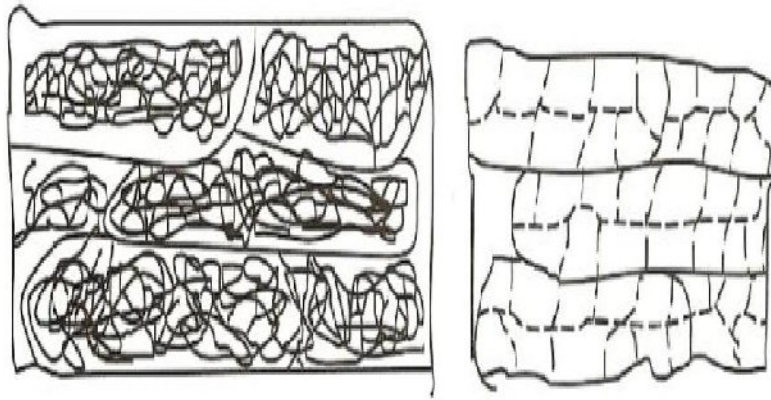


Figure 2.2: Example of recovery microstructure. [5]

2.1.2 Recrystallization

When a cold worked metallic material is heated above a certain temperature, rapid recovery eliminates residual stresses and produced a polygonized dislocation structure. New small grains then nucleate at the cell boundaries of the polygonized structure, eliminating most of the dislocations. Because the number of dislocations is greatly reduced, the recrystallized metal has low strength but high ductility. The temperature at which a microstructure of new grains that have very low dislocation density appears is known as the recrystallization temperature. The process of formation of new grains by heat treating a cold worked material is known as recrystallization. [1]

2.1.3 Grain Growth

At still higher annealing temperatures, both recovery and recrystallization occur rapidly, producing a fine recrystallized grain structure. If the temperature is high enough, the grains begins to grow with favored grains consuming the smaller grains. This phenomenon called grain growth is driven by the reduction in grain boundary area. [1]

2.2 COMPRESSION TEST

The compression test is a method for determining behavior of materials under crushing loads. Specimen is compressed and deformation at various loaded is recorded. Compressive stress and strain are calculated and plotted as a stress-strain diagram which is used to determine elastic limit, proportional limit, yield point, yield strength and compressive strength. [3]

2.3 ENGINEERING STRESS AND STRAIN

The results of a compression test apply to all sizes and cross-sections of specimens for given material if we convert the force to stress and the distance between gage marks to strain.

2.3.1 Engineering Stress

$$\sigma = \frac{F}{A_o}$$

Where;

F = instantaneous load applied perpendicularly to the specimen cross section.

A_o = the original cross sectional area before any load is applied

2.3.2 Engineering Strain

$$\epsilon = \frac{(l_i - l_o)}{l_o}$$

Where;

l_o = original length before any load applied

l_i = the instantaneous length

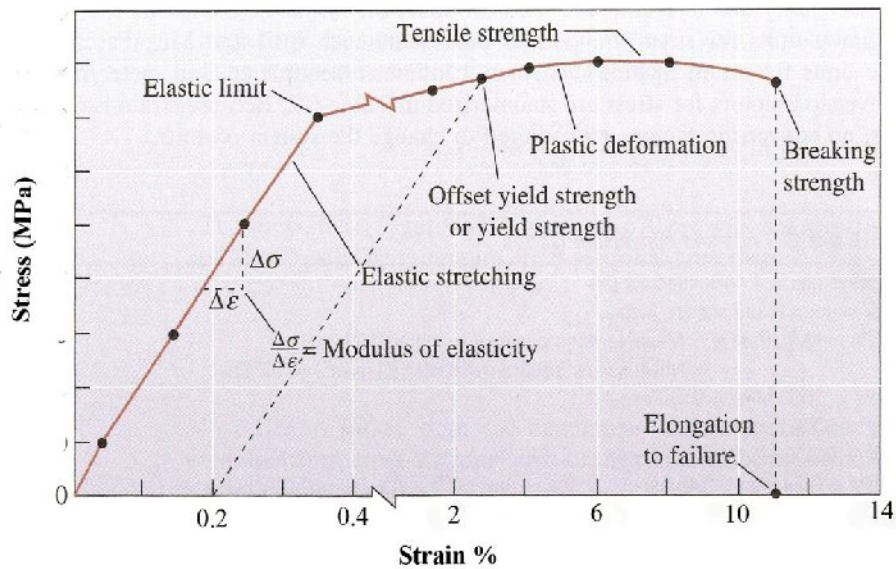


Figure 2.3: Engineering stress-strain diagram. [1]

2.4 PROPERTIES OBTAINED FROM COMPRESSION TEST

2.4.1 Yield Strength

As we apply stress to a material, the material initially exhibits elastic deformation. The strain that develops is completely recovered when the applied stress is removed. However, as we continue to increase the applied stress the materials begins to exhibit both elastic and plastic deformation. The material eventually yields to the applied stress. The critical stress value needed to initiate plastic deformation is defined as the elastic limit of the material. The proportional limit is defined as the level of the stress above which the relationship between stress and strain is not linear. [1]

In most materials the elastic limit and proportional limit are quite close. However, neither the elastic limit nor the proportional limit values can be determined precisely. Measured values depend on the sensitivity of the equipment used and defined as offset strain value (typically is 0.2%). Then, draw a line starting with this offset value of strain and draw a line parallel to the linear portion of the engineering stress-strain curve. The stress value corresponding to the intersection of this line and

the engineering stress-strain curve is defined as the offset yield strength, also often stated as yield strength.

2.4.2 Ultimate Tensile Strength

The ultimate tensile strength is the maximum strength reached in the engineering stress-strain curve. If the specimen develops a localized decrease in cross-sectional area (commonly called necking), the engineering stress will decrease with further strain until fracture occurs since the engineering stress is determined by using the original cross-sectional area of the specimen. The more ductile a metal is, the more the specimen will neck before fracture and hence the more the decrease in the stress on the stress-strain curve beyond the maximum stress.

The ultimate tensile strength of a metal is determined by drawing a horizontal line from the maximum point on the stress-strain curve to the stress axis. The stress where this line intersects the stress axis is called the ultimate tensile strength or sometimes just the tensile strength. The ultimate tensile strength is not used much in engineering design for ductile alloys since too much plastic deformation takes place before it is reached. However, the ultimate tensile strength can give some indication of the presence of defects. If the metal contains porosity or inclusions, these defects may cause the ultimate tensile strength of the metal to be lower than normal. [3]

2.4.3 Percent Elongation

The amount of elongation that a tensile specimen undergoes during testing provides a value for the ductility of the metal. Ductility of the metals is most commonly expressed as percent elongation, starting with a gage length usually of 5.1cm. In general, the higher the ductility (the more deformable the metal is), the higher the percent elongation is.

The percent elongation at fracture is of the engineering importance not only as a measure of ductility but also as an index of the quality of the metal. If porosity or inclusions are present in the metal or if damage due to overheating the metal has

occurred, the percent elongation of the specimen tested may be decreased below normal. [3]

2.4.4 Modulus of Elasticity

In the first part of tensile test, the material is deformed elastically. That is, if the load on the specimen is released, the specimen will return to its original length. For metals the maximum elastic deformation is usually less than 0.5 percent. The modulus of elasticity is related to the bonding strength between the atoms in metal and alloy. Metals with high elastic modules are relatively stiff and do not deflect easily. In the elastic region of the stress-strain diagram, the modulus does not change with increasing stress. [3]

2.4.5 Percent Reduction in Area

The ductility of a metal or alloy can also be expressed in terms of the percent reduction in area. The percent reduction in area, like the percent elongation, is measure of the ductility of the metal and is also an index of quality. The percent reduction in area may be decreased if defects such as inclusions or porosity are present in the metal specimen. [3]

2.5 MATERIAL

Table 2.1: Mechanical and physical properties of Aluminum alloy AA1100.

Properties	Value
Yield strength	103MPa
Ultimate strength	110MPa
Elongation at break	12%
Modulus of elasticity	69GPa
Melting point	540.6°C – 643°C
Annealing temperature	413°C
Recovery temperature	150°C

2.6 FRIEDEL'S MODEL

From the static recovery process, we can model the degree of recovery using Friedel's model equation. [8]

$$X_{rec} = \frac{\sigma_m - \sigma_r}{\sigma_m - \sigma_o}$$

Where;

X_{rec} = degree of recovery

σ_r = the yield stress of the recovered crystal

σ_m = the yield stress of the as deformed crystal

σ_o = the yield stress of undeformed crystal

The relationship between the degree of recovery, time and temperature was found as:

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$

Where;

X_{rec} = degree of recovery

C_1 = constant

t = time

R = gas constant (8.314 J/mol.K)

T = temperature (K)

Q = activation energy (kJ/mol)

The Friedel's model equation and for all the relationship between degree of recovery, X_{rec} time and temperature will apply to experimental result. This relationship is use to find the activation energy, Q from the graph.

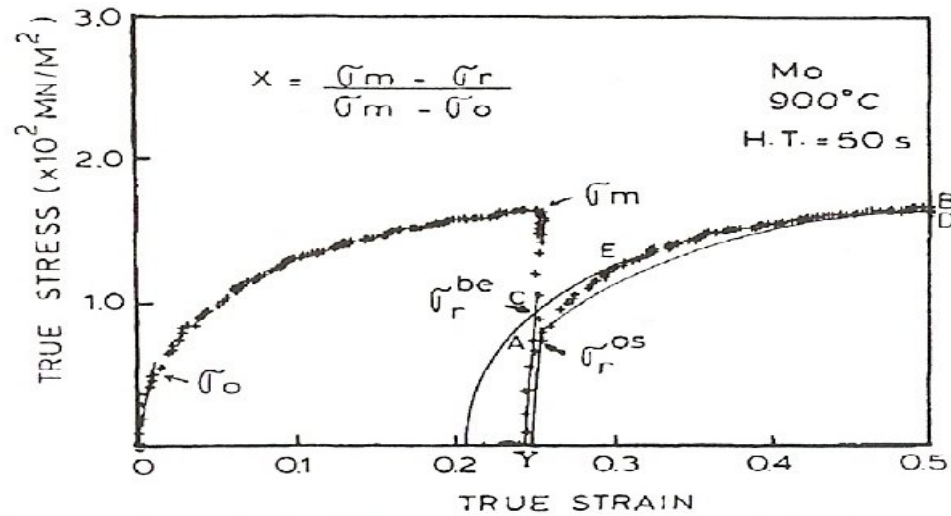


Figure 2.4: Determination of reloading flow stress and degree of softening by the back extrapolation (be) and offset (os). [8]

2.7 REVERSE CALCULATION FROM PUBLISHED RESULT

From the journal E. Nes, *Acta metall. metar. Recovery Revisited* 43 (1994), the Friedel's model equation can be applied at graph X_{rec} Vs Time to find the value of activation energy, Q .

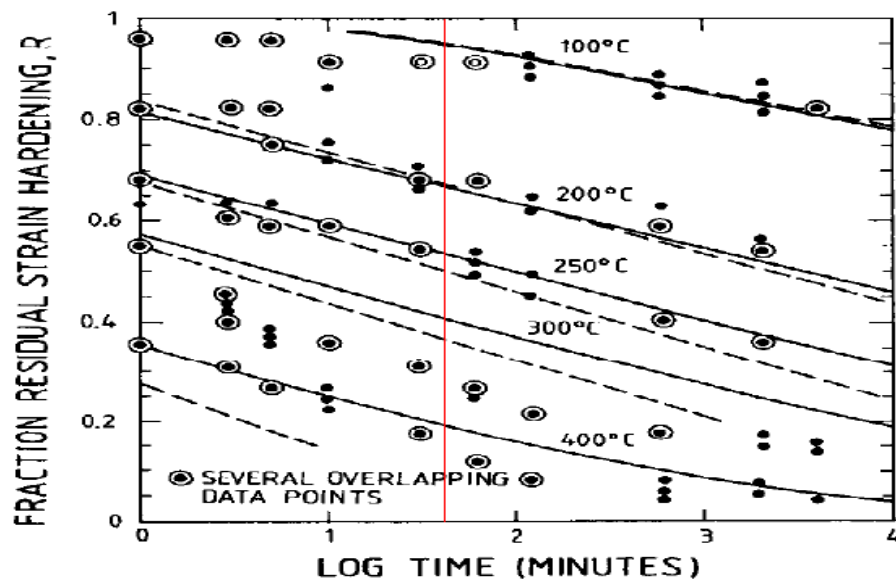


Figure 2.5: Graph X_{rec} vs time with a different temperature. [9]

To find the value of activation energy, Q the value of recovery can be measured at constant time (1 hour at red line) with a different temperatures (100°C, 200°C, 300°C and 400°C). Then, the graph X_{rec} vs $1/T$ can be plotted.

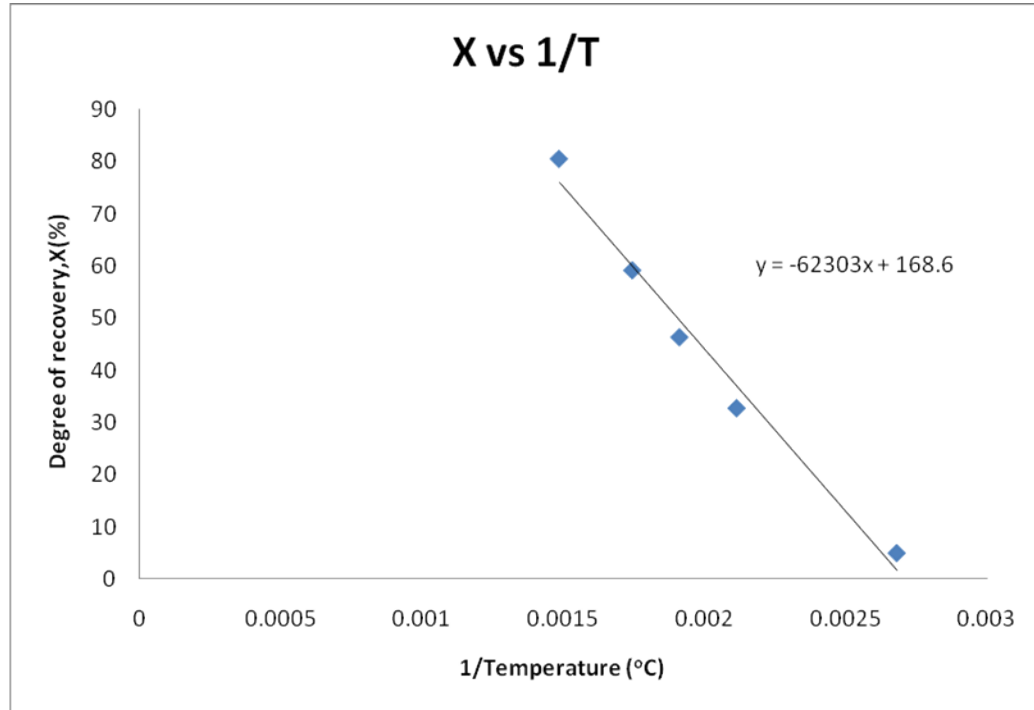


Figure 2.6: Graph X_{rec} vs $1/T$ emperature.

From graph X_{rec} vs $1/T$, the linear equation are plotted which is $y = -62303x + 168.6$. Then using Friedel's model equation, the comparison can be made to find the activation energy, Q .

$$y = mx + c$$

$$y = -62303x + 168.6 \quad (1)$$

Applied Friedel's model equation:

$$X_{rec} = C_1 \ln t - \frac{Q}{RT} \quad (2)$$

Then, made the comparison between equation (1) & (2) to find activation energy, Q:

$$\frac{-Q}{R} \left(\frac{1}{T} \right) = -62303x$$

$$\frac{-Q}{R} = -62303$$

$$\begin{aligned} Q &= 62303 \times (8.314 \text{ J/mol.K}) \\ &= 517.987 \text{ kJ/mol.} \end{aligned}$$

Finally, this Q value from the literature review will used to compare with experimental Q value.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, detailed process about static recovery is presented. Start with machining the specimens, annealing process and continue with compression test. In annealing process we go through a different test which is against pre-strain, temperature and time. Then the result from the compression test will use to validate the Fridel's model.

3.2 MACHINING PROCESS

Before we go through to a compression test, the specimens are started to be made. The specimens needed in this project have a cylinder shape and must be made around 50 specimens. The dimension for the diameter is 10mm while 25mm for the length. To make this specimens, the aluminum material will go through a few machining process.

3.2.1 Bandsaw

The raw material for aluminum alloys has a long dimension in length, so the bandsaw machine is using to cut the material with desire dimension. The material that we cut must have a tolerance (around +3mm), so the end of material can be facing using a lathe machine.



Figure 3.1: Mechanical bandsaw

3.2.2 Lathe Machine

A lathe is a machine tool which turns cylindrical material, touches a cutting tool to it, and cuts the material. The lathe is one of the machine tools most well used by machining.



Figure 3.2: Mechanical lathe machine

There are some processes that involve during the specimen which is centre drill, turning and facing.

1. Centre drill

- Applied at both end surface of specimen to hold the work piece from bending and vibrate.

2. Turning

- This is a process to reduce the raw material dimension to become 10mm diameter of specimens.

3. Facing

- This is a process to make a flat surface at the end of work piece and the tolerance that we have at specimens can be cut to become our desire length which is 25mm.

3.2.2.1 Three important element

In order to get an efficient process and beautiful surface at the lathe machining, it is important to adjust a rotating speed, a cutting depth and a sending speed.

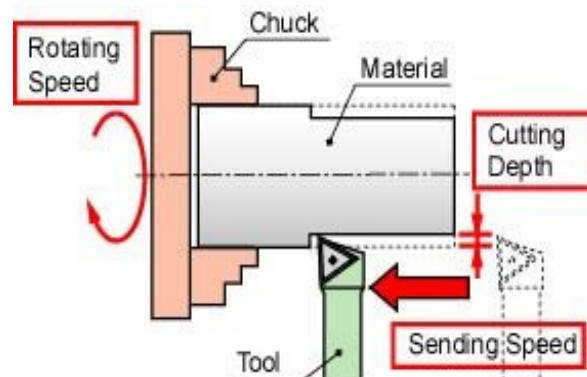


Figure3.3: Lathe machine process

1. Rotating speed

- It expresses with the number of rotations (rpm) of the chuck of a lathe. When the rotating speed is high, processing speed becomes quick, and a processing surface is finely finished. However, since a little operation mistakes may lead to the serious accident, it is better to set low rotating speed at the first stage.

2. Cutting Depth

- The cutting depth of the tool affects to the processing speed and the roughness of surface. When the cutting depth is big, the processing speed becomes quick, but the surface temperature becomes high, and it has rough surface. Moreover, a life of byte also becomes short. So, it is better to set to small value.

3. Sending speed(feed)

- The sending speed of the tool also affects to the processing speed and the roughness of surface. When the sending speed is high, the processing speed becomes quick. When the sending speed is low, the surface is finished beautiful.

3.2.2.2 Precaution steps

1. Don't keep a chuck handle attached by the chuck. Next, it flies at the moment of turning a lathe.
2. Don't touch the byte table into the rotating chuck. Not only a byte but the table or the lathe is damaged.

3.3 ANNEALING PROCESS

The next step is annealing process and all 50 specimens will undergo this process. To perform this process, the box furnace is used with using all data below:

- I. Annealing temperature: 413°C
- II. Heating rate : 5°C per minute
- III. Soaking time : 1 hour
- IV. Cooling rate : Oven cooling
- V. Heating time : $\frac{413 - \text{ambient temperature}}{5}$

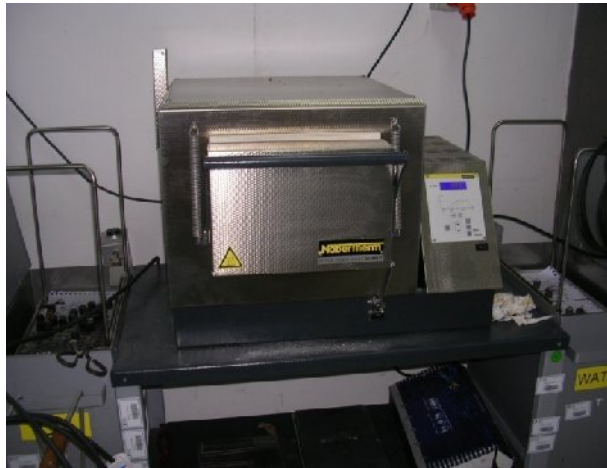


Figure 3.4: Box furnace

3.3.1 Precaution steps

1. Use glove when to put the specimen into the oven because the oven in high temperature.
2. Switch off the main switch during waiting the oven cooling.

3.4 PRE-STRAIN THE SPECIMENS

In this process, the compression test machine is used to perform pre-strain for all specimens. The yield strength and maximum strength of specimens will get from this process. In this project, the maximum load that can be applied to compression test is 50kN. So, the pre-strain value is varying with 2.5%, 5%, 7.5% and 10%. For a different temperature and time, the pre-strain value applied just 5% only. Then for each pre- strain, we are using 3 specimens to get the average value.



Figure 3.5: Compression test

3.5 RECOVERY PROCESS

This process is use to find the degree of recovery,X with different temperature, time and pre-strain.

- I. For different temperatures:
 - Varying temperature: 100°C, 150°C, 200°C and 250°C.
 - Recovery time : 1 hour
 - Heating rate : 5°C

II. For different time:

- Varying recovery time: 1 hour, 2 hour, 3 hour and 4 hour.
- Recovery temperature : 150°C
- Heating rate : 5°C

III. For different pre-strain:

- Recovery temperature: 150°C
- Recovery time : 1 hour
- Heating rate : 5°C

3.6 PRE-STRAIN RECOVERED SPECIMENS

In this process, the recovered specimens will pre-strain again to find the new yield strength. The new yield strength is used to find degree of recovery, X_{rec} and all the data will recorded using compression test machine. For a different pre-strain, varying pre-strain with 2.5%, 5%, 7.5% and 10%. For a different temperature and time, just 5% pre-strain was applied.

3.7 ANALYZING DATA

After collected the data such as yield strength, maximum strength and recovery yield strength, we can find X_{rec} using equation below:

$$X_{rec} = \frac{\sigma_m - \sigma_r}{\sigma_m - \sigma_o}$$

After that, we can plotted the graph using excel to find the relationship between degree of recovery, X_{rec} with pre-strain, temperature and time.

3.8 APPLYING FRIEDEL'S MODEL

From the graph that we plotted, we can find the activation energy, Q using relationship of equation below:

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$

3.9 COMPARISON

After we get the activation energy, Q from the plotted graph, we can compare the Q value with a theory value of activation energy. From the comparison, we can conclude whether we can validate the static recovery process using Friedel's model or not.

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

In this chapter, the result of relationship between degree of recovery, X_{rec} with pre-strain, temperature and time are shown. The calculation of how to find the activation energy, Q also will produce step by step. Then, some discussion is made from the result to prove this project objective.

4.2 RESULT

From this experiment, 3 graphs can be plotted which is:

1. Degree of recovery, X_{rec} versus $1/\text{Temperature}$ at constant pre-strain 5% and time for 1 hour.
2. Degree of recovery, X_{rec} versus $\ln t$ at constant pre-strain 5% and temperature for 150°C.
3. Degree of recovery, X_{rec} versus pre-strain at constant time for 1 hour and temperature for 150°C.

4.2.1 Relationship between X_{rec} vs $1/\text{Temperature}$

The relationship between degree of recovery, X_{rec} and temperature can be plotted using Friedel's model equation. The data below has shown the characteristic of softening before and after static recovery process using pre-strain 5% at different temperature.

Table 4.1: Data for temperature 100°C at 5% and 1 hour

σ_o	σ_m	σ_r	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_o$	$1/T$	X_{rec}	$X_{\text{rec}} (\%)$
41.141	112.1	104.96	7.14	70.959	0.002681	0.100621	10.0621
39.5	111.2	106.2	5	71.7	0.002681	0.069735	6.9735
41.62	111.8	93.78	18.02	70.18	0.002681	0.256768	25.6768

Table 4.2: Data for temperature 150°C at 5% and 1 hour

σ_o	σ_m	σ_r	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_o$	$1/T$	X_{rec}	$X_{\text{rec}} (\%)$
22.83	112.9	97.199	15.701	90.07	0.002364	0.17432	17.432
43.49	110.7	97.72	12.98	67.21	0.002364	0.193126	19.3126
58.42	107.3	96.711	10.589	48.88	0.002364	0.216633	21.6633

Table 4.3: Data for temperature 200°C at 5% and 1 hour

σ_o	σ_m	σ_r	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_o$	$1/T$	X_{rec}	$X_{\text{rec}} (\%)$
46.9	113	77.558	35.442	66.1	0.002114	0.536188	53.6188
64.65	110.5	70.59	39.91	45.85	0.002114	0.870447	87.0447
45.11	111.9	68.64	43.26	66.79	0.002114	0.647702	64.7702